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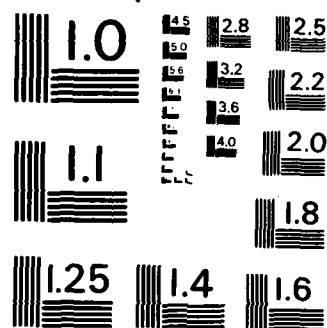
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STUDY OF FUEL DETERIORATION AND ADDITIVE-INHIBITION

INTERIM REPORT
BFLRF No. 193

By

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Belvoir Fuels and Lubricants Research Facility (SwRI)
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San Antonio, Texas

Under Contract to

**U.S. Army Belvoir Research
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FOREWORD

This report was prepared at the U.S. Army Fuels and Lubricants Research Laboratory (USAFLRL) located at Southwest Research Institute, San Antonio, TX, under Contract Nos. DAAK70-82-C-0001 and DAAK70-85-C-0007, for the period 1 October 1983 through 30 September 1984. Work was funded by the U.S. Army Belvoir Research and Development Center, Ft. Belvoir, VA, with Mr. F. W. Schaekel (STRBE-VF) serving as contracting officer's representative. Project technical monitor was Mr. M. E. LePera, STRBE-VF.

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I. INTRODUCTION

The stability of middle distillate fuels during storage is an important consideration in both commercial and military fuel supply systems. Deterioration products can cause gum-like deposits in fuel supply systems which can, in turn, lead to filter and injector nozzle plugging.(1)* The requirement for long-term storage stability of military fuels is critical in the prepositioning of fuel supplies where good fuel quality must be maintained.(2)

During the past several years, numerous fuel-related problems have surfaced within U.S. Army facilities.(3) These problems are likely to grow with increased use of heavy crudes, variations in refining techniques, and use of synthetic fuels. Fuel storage stability is also dependent on other factors, such as fuel composition, prior history, additive treatment, container design and/or composition, and environmental conditions.

The reactions taking place within finished fuels and the products formed from these reactions have been studied for many decades. It was pointed out over half a century ago that the presence of di-olefins in cracked distillates were responsible for gum formation.(4) Other works written in the late 1920's and 1930's verify and expand on these findings. A postulation as to the chemical character of gums from heating oils was made approximately a quarter of a century ago.(5) Elemental concentration and infrared and mass spectrometric analyses indicated that these particular sediments were esterified, condensed molecules containing nitrogen heterocycles.

It has been further suggested that sediment-formation proceeds by (1) the oxidation of "reactive hydrocarbons", nitrogen heterocycles, and sulfur heterocycles to their corresponding hydroperoxides, followed by (2) decomposition of these hydroperoxides to aldehydes, then (3) reaction of these aldehydes with other hydroperoxides to form peroxyhemiacetals, which (4) condense, forming insoluble polymers.

* Underscored numbers in parentheses refer to the list of references at the end of this report.

More recent work, in which sediment formation was promoted by adding 2,5-dimethylpyrrole (DMP) to pure hydrocarbons containing no sulfur, as well as to real fuels, has led to the postulation of several structural possibilities for the sediment material.(6)

The sediment-promoting properties of 5-ethyl-2-methylpyridine and 2,5-dimethylpyrrole were examined in separate experiments in which they were added to acid-washed fuel at the 50-ppm level.(7) Sediment formation and peroxide concentrations were increased 20-fold by the addition of the pyridine compound when compared to the pyrrole compound.

Similar results were obtained after producing a stable diesel fuel (Texaco D 454) by removing the polar constituents with silica gel.(8) Fractions were eluted from the silica gel with hexane, toluene, and tetrahydrofuran (THF). After evaporating the solvents, each of the resulting extracts was added to an aliquot of the stabilized fuel. Sediment formation was monitored under storage conditions. Only the THF extract produced substantial sediment formation. Shale-derived jet fuel (Shale I) can be stabilized by acid extractions of the nitrogen bases.(7) Replacing these extracted nitrogen compounds and subsequent aging caused the fuel to form sediments. Also, increased peroxide levels were found in the aged fuel. Mass spectral analysis of the extracted compounds showed the majority to be substituted pyridines and quinolines. Lesser amounts of indoles, pyrroles, and piperidines were also seen.

In other work, soluble sediment precursors from No. 2 home heating oil were characterized by field ionization mass spectrometry.(9) Molecular weight profiles were obtained for residues from fresh and aged fuels which had been concentrated under nitrogen. Increased levels of higher molecular weight components were observed with increased aging. Initially, dimers (~400 MW) are formed, and as aging progresses, the concentration of trimers (~600 MW) was observed to increase.

In addition, it has been demonstrated that certain compounds which do not produce sediment themselves (e.g., isoquinoline) do enhance the activity of other sediment promoters (e.g., dimethylpyrrole).(10)

Recent work at the Naval Research Laboratory (NRL) has produced information on both peroxide formation (11-13) and light scattering (14) in aging DFM spiked with DMP.

The above work and, indeed, virtually all the pertinent literature had addressed the chemistry of particulate and adherent insolubles formation. This was also a goal of the FY82-83 portion of this program. These data were presented at Army Research Office Engine/Fuels Workshops (15,16) and at the Long-Term Storage Stabilities of Liquid Fuels Conference. (17) Interim Report AFLRL No. 168 (18) summarized activity in fuel stability research and included a historical background and bibliography.

The FY84 research effort addressed an area that has been discussed extensively but has generated minimal research activity. That area is concerned with the actual physical growth of insoluble particulate matter and its relationship to adherent insolubles formation.

Of primary interest during FY84 was the study of the kinetics of insoluble particulate formation as a function of temperature and fuel type. This was accomplished through determination of particle size and number relationships as well as filterable weight by ASTM D 2276. A second objective was to examine the potential correlation between gravimetrically determined filterable particulate weight and a mathematical determination using measured particle size and number.

The particle weight per unit volume was calculated from the formula for a sphere, using an assumed particle density. The form of the equation was

$$W = \sum_{n=1}^X N_n \frac{4}{3} \pi r_n^3 d \quad (1)$$

where W is the weight per unit volume of the particles

X is the number of size ranges studied

N_n is the number of particles per unit volume counted in size range n

r_n is the mean radius in size range n

d is the particle density

Adherent insolubles, peroxide number, bromine number, and percent carbon, hydrogen, nitrogen, and sulfur were also measured as a function of aging time and temperature when sufficient material was available. Three fuels and four additives were used in a series of 18 experiments, which are documented in this interim report.

II. EXPERIMENTAL PROCEDURE

A. Sample Containers

Prior to each experiment, all containers were cleaned according to the ASTM D 4057 procedure. This cleaning consisted of a solvent rinse, soap and water wash, water rinse, and oven dry. In 15 of the 18 experiments, 1-liter borosilicate glass bottles were used. In the remaining three experiments, two used a 12-gallon (45-liter) Pyrex® carboy and the third used a 30-gallon (112.5-liter) metal drum.

B. Fuel Preparation and Aging

Three types of fuels were used for these experiments. Cat 1-H was used most often due to its relative instability. A petroleum-based JP-5 specifically purchased with no additives present and a referee diesel fuel containing 1 wt% sulfur were also used. Prior to aging, all fuels were passed through a 0.8- μ m mixed cellulose ester membrane filter. The borosilicate glass bottles were filled with 950 mL of fuel, the carboys were filled with 10 gallons (38 liters) of fuel, and the metal drum was filled with 25 gallons (95 liters) of fuel. Three temperatures were employed in this matrix: 65°, 80°, and 95°C. A sufficient number of borosilicate glass bottles were placed in the temperature-controlled bath to permit periodic removal in pairs throughout the experiment. The 12- and 30-gallon containers were sampled by the removal of 1-liter aliquots. Due to the large volumes of fuel used for the experiment, removal of the aliquots was considered to have an insignificant effect on the overall aging process.

C. Additives

Four additive materials were used in this work. These materials consisted of:

1. Additive-15 containing a rust inhibitor, dispersant, antioxidant, color stabilizer, and metal deactivator. Addition was made to produce a concentration of 82 ppm (25 lb/1000 bbl) in the fuel.
2. Additive-11 containing a dispersant, antioxidant, and metal deactivator. Addition was made at the same concentrations as Additive 15.
3. Single-Package Additive (SPA), a proprietary commercial material which included components similar to those listed in items 1 and 2 above plus a biocide. One mL of additive was placed in one liter of sample.
4. Additive-B, a biocide used in conjunction with Additive-15 to satisfy requirements of MIL-S-53021, was added at approximately 270 ppm.

D. Analysis

1. Filterable Particulate Weight

The quantity of fuel-insoluble particulate matter was determined by ASTM D 2276 procedures modified to use isooctane rather than petroleum ether or refrigerant 113 as the filter rinse solvent.

2. Particle Size/Number Distribution

A HIAC/Royco model PC-320 particle in liquid counter and LAS-346 laser system were used to determine particle size and number distribution in the aged fuels. Six channels were employed with some variation in size range detected by each channel as experience dictated. The final measuring ranges and chan-

nel numbers selected were 0.5-0.8 μm (Channel 1), 0.8-1.0 μm (Channel 2), 1.0-2.0 μm (Channel 3), 2.0-3.0 μm (Channel 4), 3.0-5.0 μm (Channel 5), and $>5 \mu\text{m}$ (Channel 6) apparent particle diameter.

The particle concentration was calculated using the equation:

$$\text{Concentration (mg/L)} = (0.129 N_1 + 0.344 N_2 + 1.590 N_3 + 7.363 N_4 + 30.16 N_5 + 241.274 N_6) \times 10^6 \quad (2)$$

where $N_1 - N_6$ are the number of particles counted in each channel, respectively.

The average of maximum and minimum particle radii detected by each channel was used, and the assumption of 0.9 gm/cm^3 for particle density was incorporated into the calculation of the coefficients in Equation 2.

Five of the channels used were discretely defined. However, the highest channel, Channel 6 ($>5 \mu\text{m}$), is open ended. In these studies, a mean radius of $4.0 \mu\text{m}$ was assumed. However, further work is required to determine the accuracy that this value represents.

3. Peroxide Number

The peroxide number of the fuels was determined using ASTM D 3703.

4. Bromine Number

The bromine number of the fuels was determined using ASTM D 2710.

5. Adherent Insolubles

After removal of the aged fuel from the containers, the interior surfaces were rinsed with heptane to remove residual fuel, followed by a trisolvent rinse composed of equal volumes of toluene, acetone, and methanol (TAM). The TAM rinsings were then placed in a tared beaker and allowed to evaporate to dryness at 150°C in an airjet apparatus (as used in ASTM Method D 381). The

weight of residue was determined in duplicate and reported as adherent insolubles per 100 mL of fuel.

6. Elemental Analysis

a. Carbon/Hydrogen - Total carbon and total hydrogen were analyzed gravimetrically. Samples placed in a horizontal Linberg oven were converted to CO_2 and H_2O which were absorbed on preweighed substrates. The difference in weight is used to calculate percent carbon and percent hydrogen.

b. Nitrogen - Total nitrogen was determined using an Antek model 720/771 pyrochemiluminescence apparatus.

c. Sulfur - Total sulfur was determined using an EDAX X-ray fluorescence spectrometer. Use of this nondestructive technique allows further analyses to be performed on the sample.

E. Experimental Matrix

An overall summary of the experimental work described previously is given in Table 1.

III. EXPERIMENTAL RESULTS AND DISCUSSION

A. Petroleum JP-5

Three experiments were conducted using JP-5 without additives. The first two experiments were conducted less than 1 month apart and were designed to show variation due to aging at 80° and 95°C. Measured and calculated particulate weight, adherent insolubles, peroxide number, and nitrogen content of the gums, were determined for both experiments. Bromine number was determined for the 95°C test, and sulfur concentration of the gums was determined for the 80°C test.

TABLE 1. FY84 EXPERIMENTAL MATRIX

Fuel	Aging Temp, °C	Container	Duration, Days	Determi- nations*	Additives
JP-5 (raw)	95	1-L Pyrex®	36	A-C	None
	95	1-L Pyrex®	21	A-F	None
	80	1-L Pyrex®	42	A-D, F, G	None
High-Sulfur	95	1-L Pyrex®	14	A-G	None
Diesel	95	1-L Pyrex®	14	A-I	Additive-15
Cat 1-H	95	1-L Pyrex®	5	A-D, F, G	Additive-11
Cat 1-H	80	1-L Pyrex®	39	A-D, F, G	Additive-11
Cat 1-H	65	1-L Pyrex®	32	A-D, F, G	Additive-11
Cat 1-H	95	1-L Pyrex®	7	A-D, F	None
Cat 1-H	95	1-L Pyrex®	4	A-C	None
Cat 1-H	80	12-Gal. Pyrex®	23	A-C, F-I	None
Cat 1-H	80	12-Gal. Pyrex® & 30-Gal. Drum	21	A, B	None
Cat 1-H	80	1-L Pyrex®	14	A-C	None
Cat 1-H	80	1-L Pyrex®	23	C	None
Cat 1-H	65	1-L Pyrex®	32	A-D, F, G	None
Cat 1-H	65	1-L Pyrex®	25	A-C	None
Cat 1-H	80	1-L Pyrex®	14	A-D	Additive-15 + Additive-B
Cat 1-H	80	1-L Pyrex®	14	A-D	SPA, Commercial Single-Package Additive

* A Filterable particulate weight
 B Particulate size/number distribution
 C Adherent insolubles weight
 D Peroxide number
 E Bromine number

F Nitrogen content
 G Sulfur content
 H Carbon content
 I Hydrogen content

The third experiment was designed to determine if bottle location within a constant temperature air oven would significantly change the result.

In both the 80° and 95°C experiments, induction periods were noted for both particulate and adherent materials. These periods were 8 and 3 days for particulates and 15 and 4 days for the adherents, respectively, thus indicating particulate formation prior to adherent formation in both cases. The results of these experiments may be seen graphically in Figures 1 and 2. Calculated particulate weight results are consistently lower than the gravimetric data; however, the curve shapes reflect a strong similarity in the data trends.

The behavior of peroxide number (Figure 3) appears to be rather erratic, with the 95°C test eventually exhibiting lower values than the 80°C test. The maximum bromine number was 0.15 at 21 days. Nitrogen and sulfur analyses and the average values for each are shown in Table 2. Reference 12 reports similar tests at 100°C for both petroleum- and shale-derived JP-5. Although the peroxide number results from petroleum JP-5 testing in this laboratory (maximum value 97 ppm) show good agreement with the shale fuel results reported in Reference 12, the petroleum JP-5 results do not show good agreement. The petroleum JP-5 results in Reference 12 indicated a maximum value of 8 ppm.

A schematic diagram of the air bath bottle locations in another experiment with JP-5 is shown in Figure 4. An obvious trend in apparently high adherent insolubles weight follows the middle row and right rear samples (bottles B, E, H, I). Three of these four samples were also the only ones to exhibit filter plugging in the ASTM D 2276 test.

Thermocouple placement did not indicate undue temperature fluctuations at these points; therefore, light through the vent opening may be an explanation for these results. The vent has since been rerouted to prevent external light from entering the heating chamber.

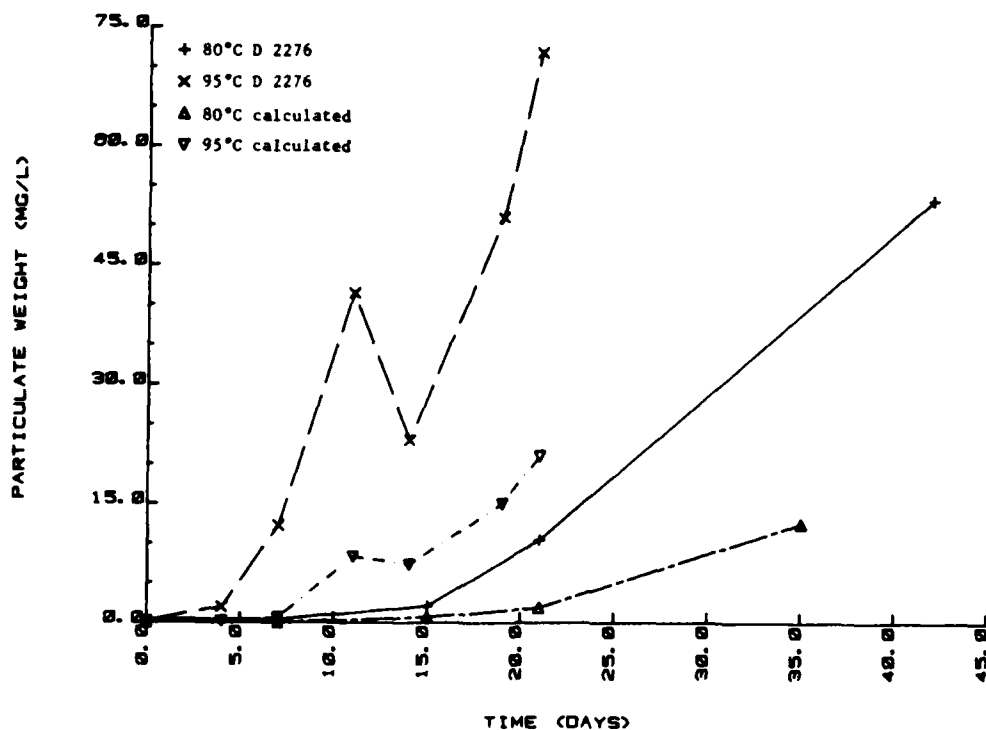


FIGURE 1. COMPARISON OF CALCULATED AND MEASURED PARTICULATE WEIGHT FOR ADDITIVE-FREE JP-5

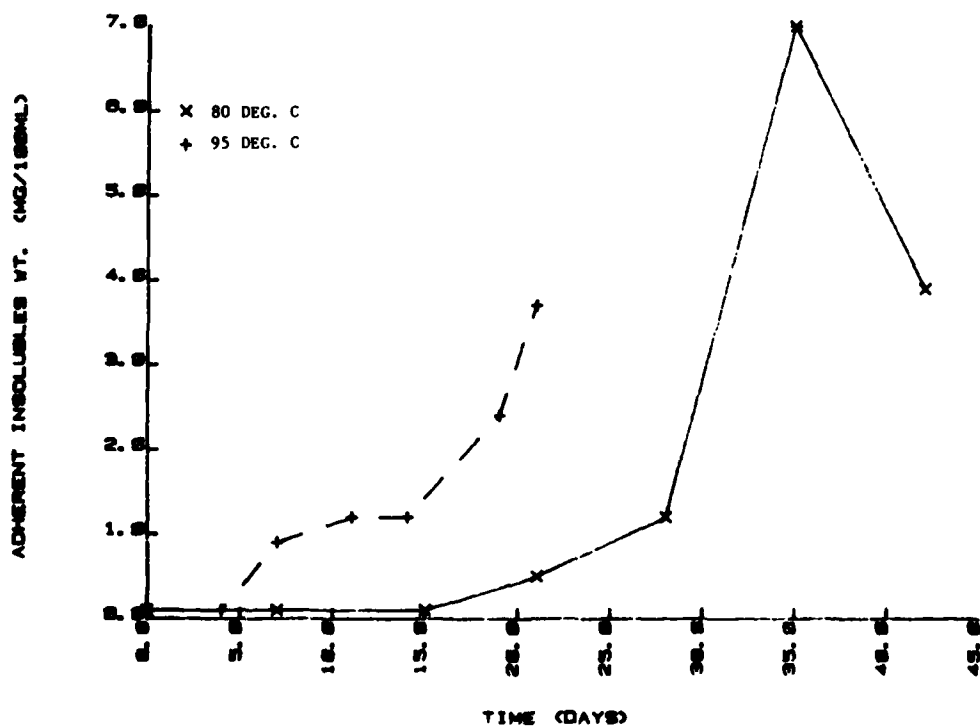


FIGURE 2. ADHERENT INSOLUBLES FORMATION AS A FUNCTION OF TIME FOR ADDITIVE-FREE JP-5

TABLE 2. ELEMENTAL COMPOSITION OF ADHERENT
INSOLUBLES FROM NEAT JP-5

Day	80°C		95°C
	%N	%S	%N
4			2.9
7			2.6
11			2.4
14			2.7
19			2.4
21	3.1		3.1
28	3.7		
35	2.6	2.8	
42	1.9	1.3	
Average	2.8	2.0	2.7

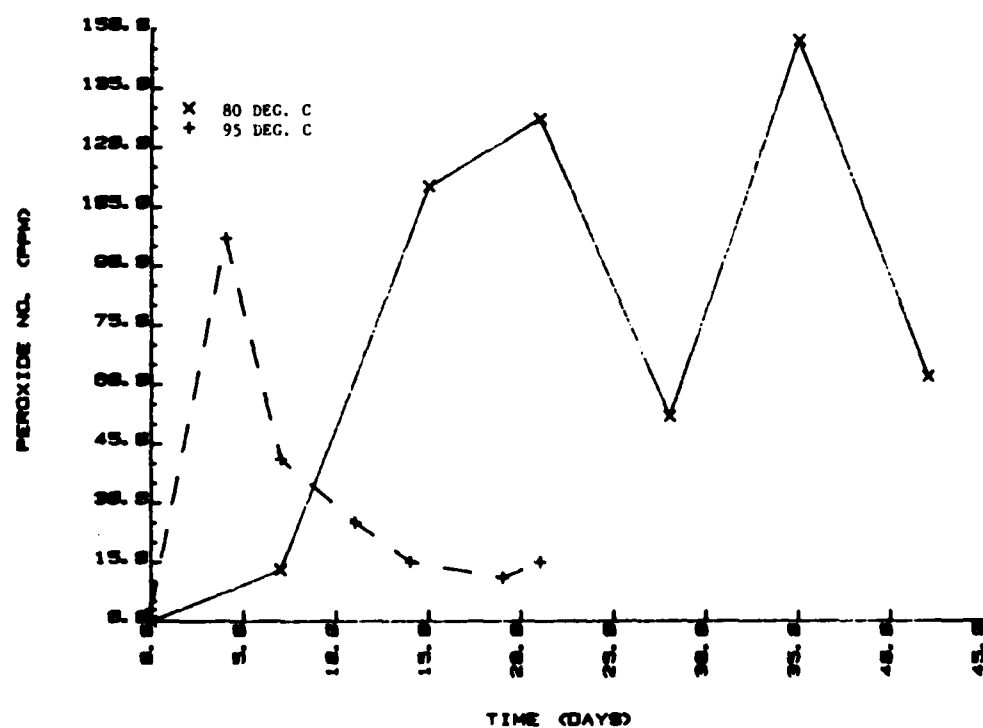


FIGURE 3. PEROXIDE NUMBER AS A FUNCTION OF TIME
FOR ADDITIVE-FREE JP-5

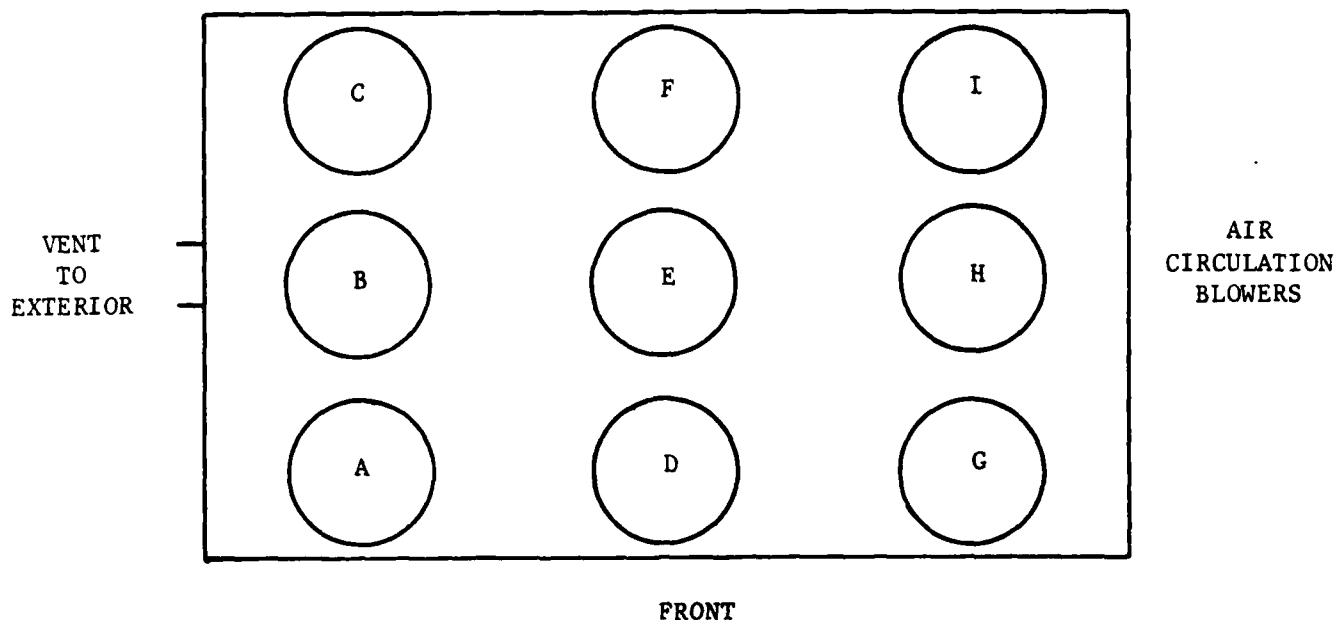


FIGURE 4. SCHEMATIC OF BOTTLE PLACEMENT IN OVEN

B. Referee Diesel Fuel Containing 1% Sulfur

At 95°C, two experiments with and without Additive-15 were conducted. ASTM D 2276 (filterable particulate weight), calculated particulate weight, adherent insolubles weight, peroxide number, bromine number, and N and S concentrations were determined for each test. The C and H content of the adherent materials was also determined for the additive-containing fuel.

The D 2276 values for the additive-treated fuel were generally lower than for the nontreated fuel. The same was true for the calculated particulate weight (Figure 5). However, in both cases the calculated weights were lower than the respective D 2276 values. Peroxide and bromine numbers (Figures 6 and 7) were also lower for the additive-treated fuel. The curves for both experiments parallel each other, including a peak maximum at 4 days in the peroxide number determination. Conversely, adherent insolubles formation was greater for the additive-treated fuel (Table 3 and Figure 8), probably due to the smaller average particle sizes and thus a greater diffusion capability.

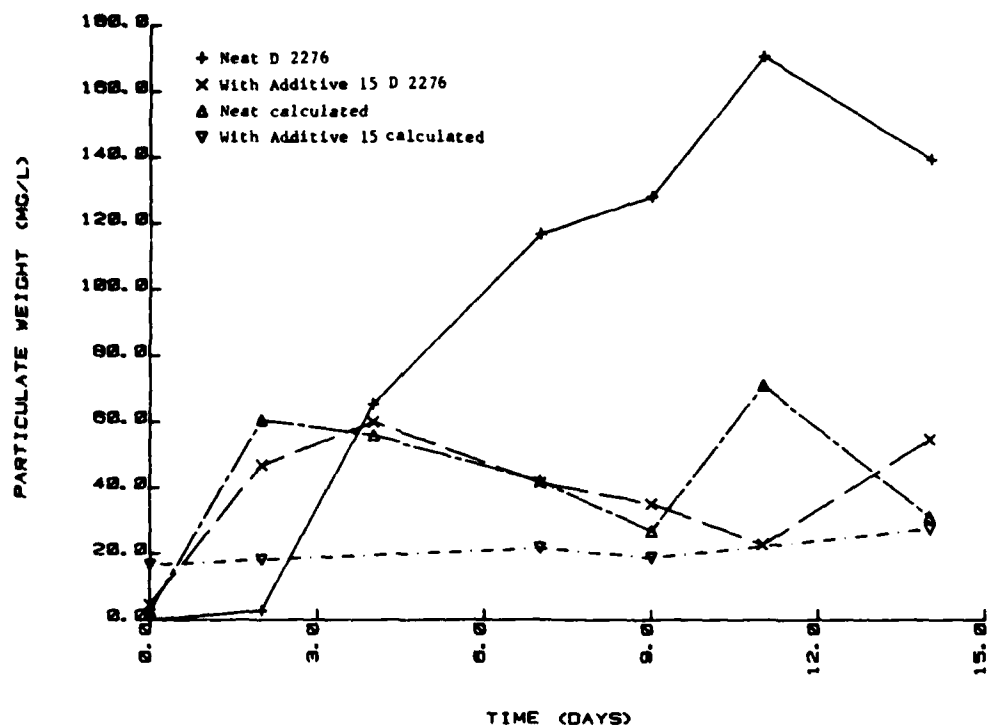


FIGURE 5. COMPARISON OF CALCULATED AND MEASURED PARTICULATE WEIGHT FOR 1% SULFUR DIESEL FUEL

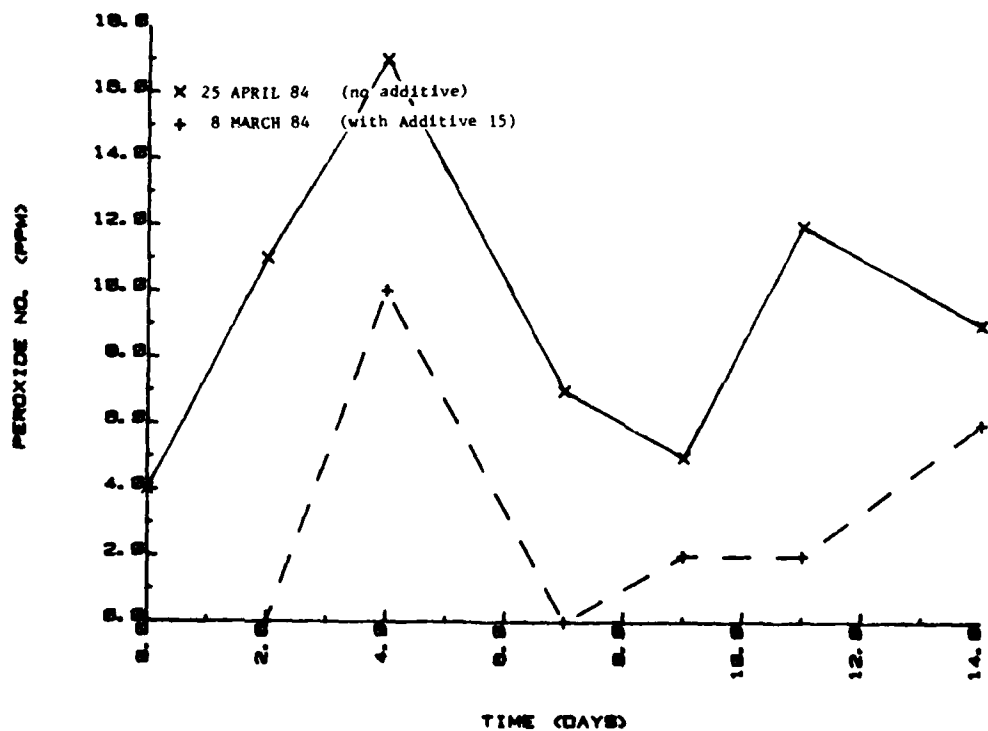


FIGURE 6. PEROXIDE NUMBER VERSUS TIME FOR 1% SULFUR DIESEL FUEL AGED AT 95°C

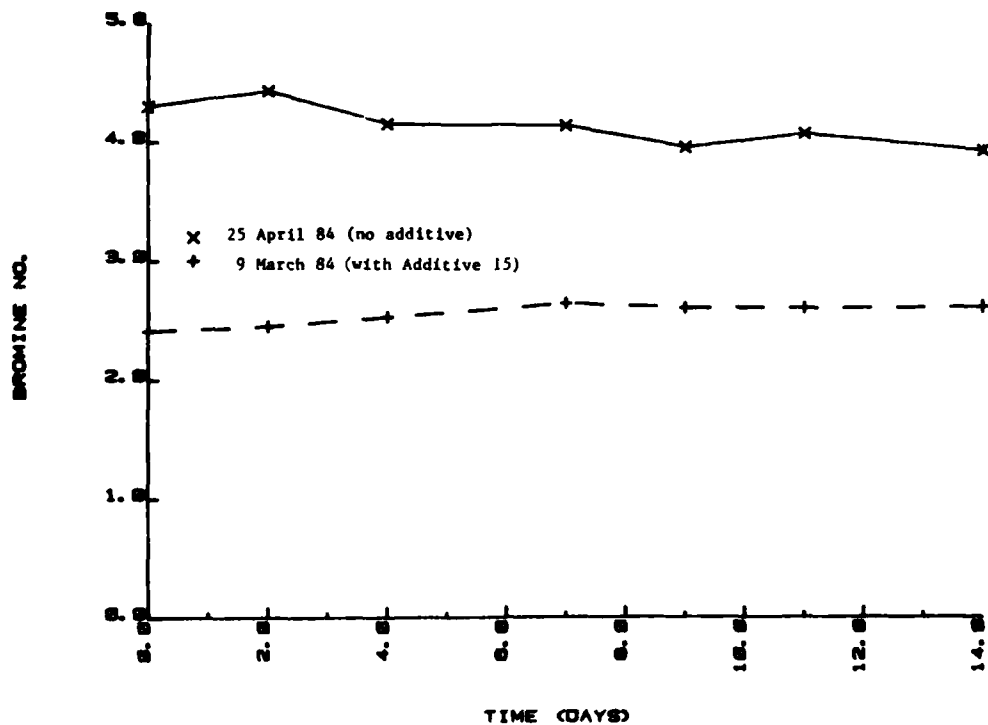


FIGURE 7. BROMINE NUMBER VERSUS TIME FOR
1% SULFUR DIESEL FUEL AGED AT 95°C

TABLE 3. HIGH-SULFUR DIESEL FUEL

Chemical Composition:

<u>Fuel</u>	<u>%C</u>	<u>%H</u>	<u>%S</u>	<u>%N</u>
Neat	--*	--	1.11	--
Additive-treated**	--	--	1.11	0.0075

Adherent Insolubles

Average

Neat	--	--	6.9	3.7
Additive-treated	67.1	6.3	7.6	4.1

* Not determined

** Fuel treated with 25 pounds of Additive-15 to 1000 barrels of fuel

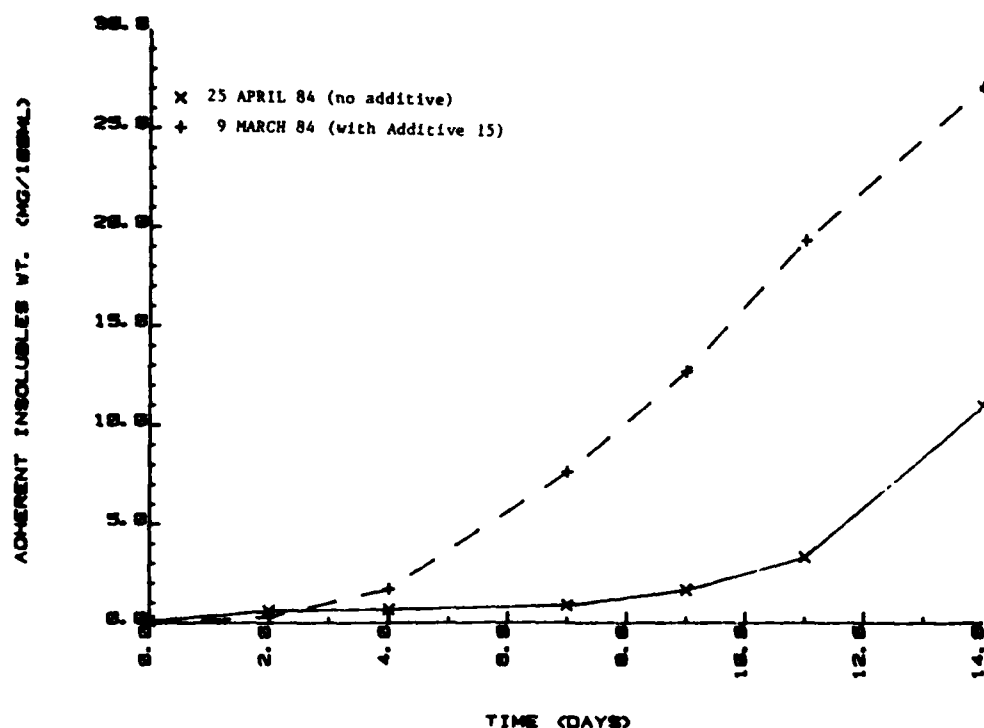


FIGURE 8. ADHERENT INSOLUBLES FORMATION VERSUS TIME FOR 1% SULFUR DIESEL FUEL AGED AT 95°C

C. Cat 1-H Fuel

1. Additive-11-Treated Cat 1-H Fuel

Started less than 6 weeks apart, the three experiments indicate differences between 65°, 80°, and 95°C storage. Analyses include filterable particulate weight by ASTM D 2276, calculated particulate weight, adherent insolubles weight, peroxide number, and N and S concentrations. Except for the elemental concentrations, the measurements made at 95°C exceeded significantly those made at 65°C. The data for 80°C were intermediate in magnitude. Peroxide numbers at 80°C were similar to those of the petroleum JP-5 at 100°C as reported in Reference 12. These data are shown in Figures 9 through 12. Values for the elemental composition of the fuel and adherent insolubles are shown in Table 4. Data for qualitatively measured and calculated particulate correlations are remarkably similar for all three temperatures. As with

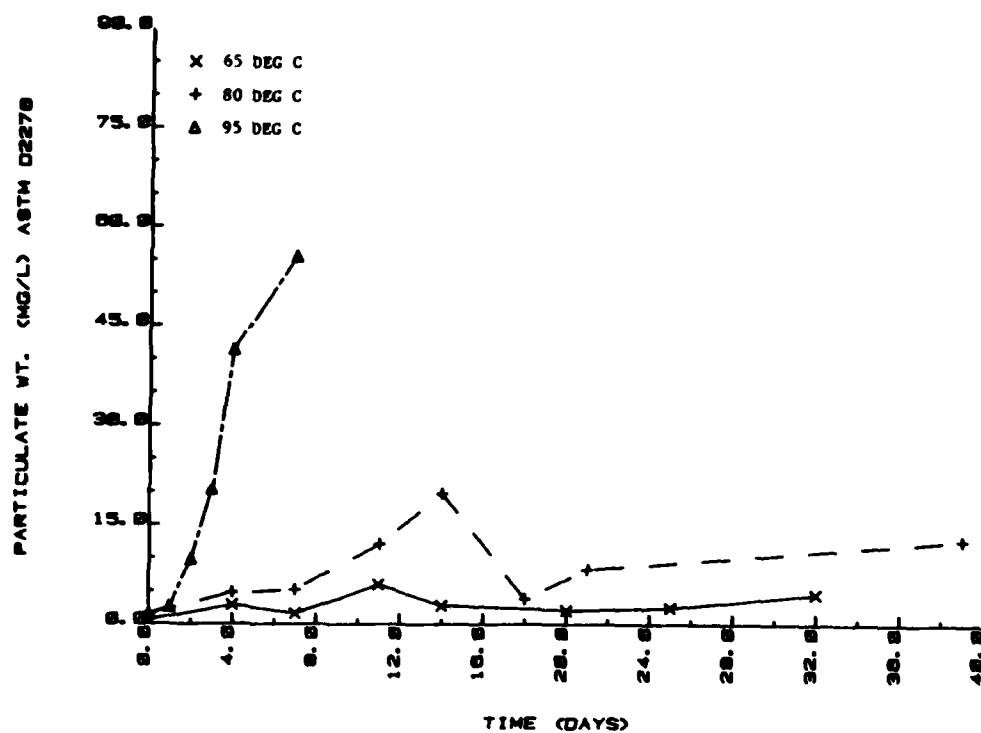


FIGURE 9. MEASURED PARTICULATE WEIGHT AS A FUNCTION OF TIME FOR CAT 1-H + ADDITIVE-11

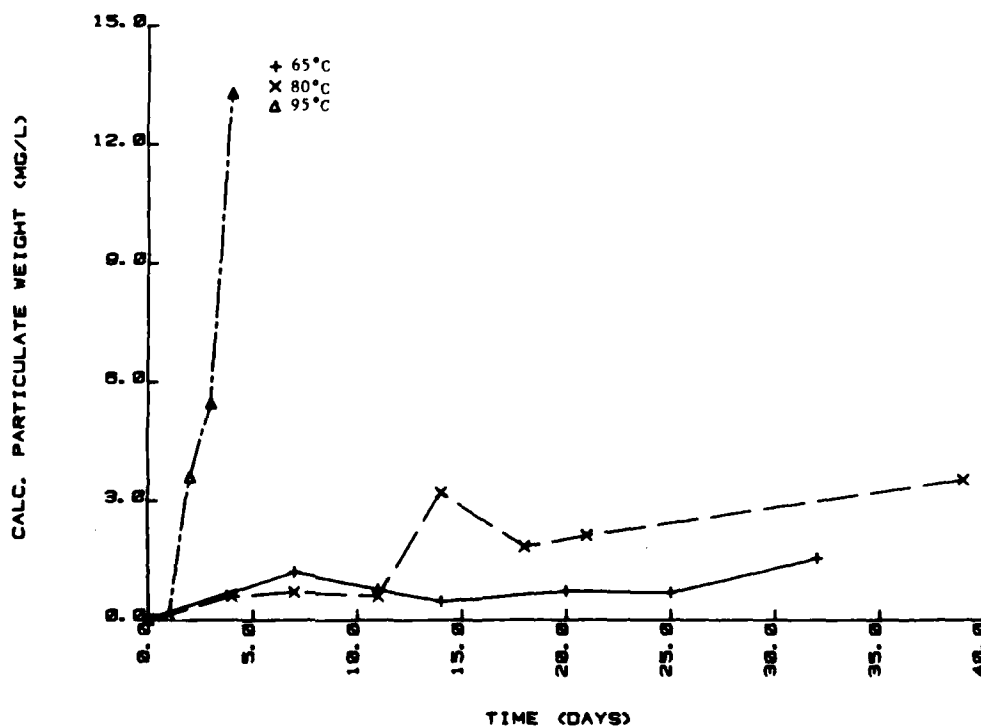


FIGURE 10. CALCULATED PARTICULATE WEIGHT AS A FUNCTION OF TIME FOR CAT 1-H + ADDITIVE-11

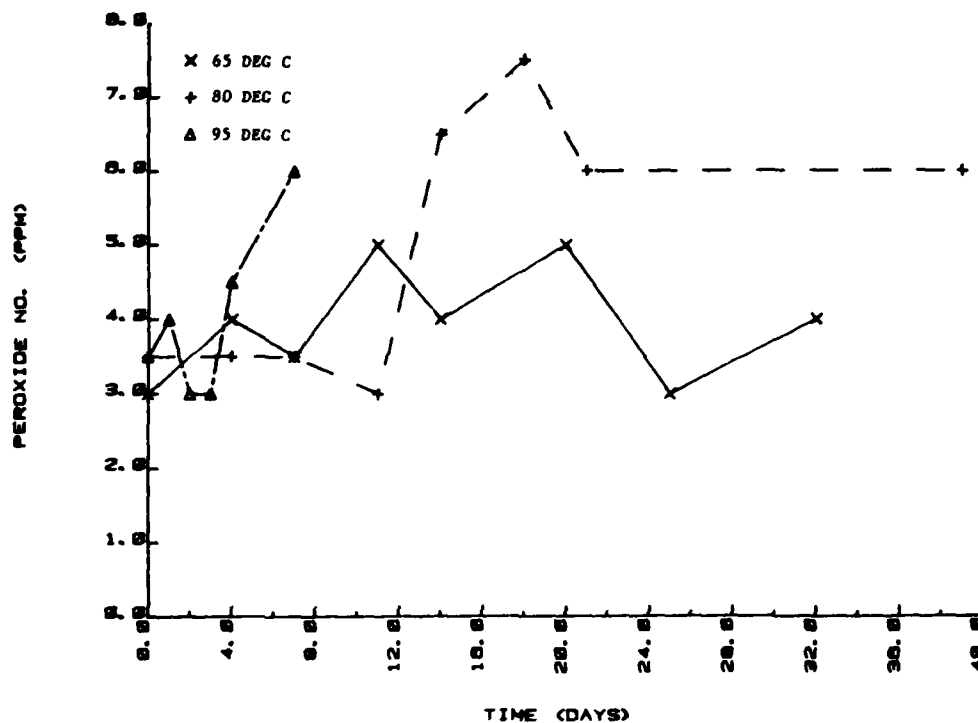


FIGURE 11. PEROXIDE NUMBER VERSUS TIME FOR CAT 1-H + ADDITIVE-11

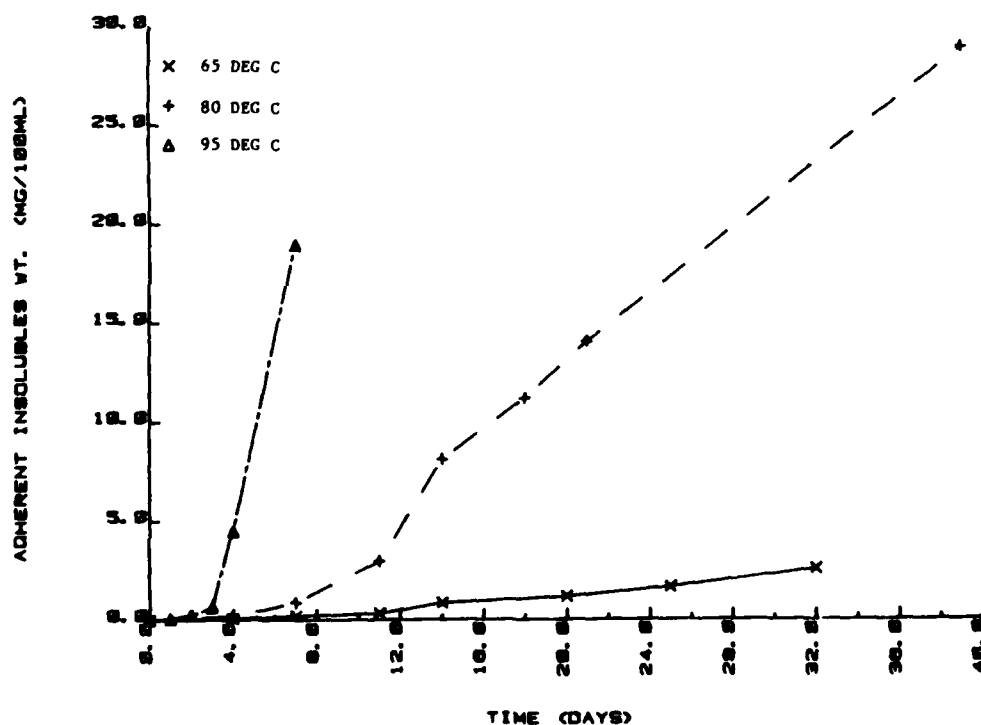


FIGURE 12. ADHERENT INSOLUBLES FORMATION AS A FUNCTION OF TIME FOR CAT 1-H + ADDITIVE-11

TABLE 4. AVERAGE CHEMICAL COMPOSITION OF CAT 1-H DERIVED MATERIALS

Sample	°C	%H	%N	%S
Neat fuel	86.35	13.04	0.005	0.42
Adherent insolubles from neat fuel				
65°C	--*	--	4.4	8.9
80°C**	64.57	6.66	4.5	7.82
95°C	--	--	3.5	8.0
Fuel + Additive-11				
65°C	--	--	3.3	15.5
80°C	--	--	3.1	9.2
95°C	--	--	3.3	9.6
Fuel + Additive-15/ Additive B				
80°C	--	--	3.8	--

All containers were 1-liter Pyrex® except as noted

* Not determined

** 12-gallon Pyrex® carboy

previous measurements, the calculated results are lower than the measured, in this case, by approximately a factor of five.

2. Neat Cat 1-H Fuel at 95°C

Analyses in these two experiments consisted of filterable particulate weight by ASTM D 2276, calculated particulate weight, and adherent insolubles weight. Peroxide number and nitrogen content were also measured for one of the experiments. These tests were run approximately 4 months apart, and they track each other quite well, indicating minimum effect of sample storage on fuel quality. Again, the calculated results are lower than the gravimetric results for particulate concentration, although qualitatively the data are consistent. These data are presented as Figures 13 and 14. Nitrogen and sulfur concentrations are shown in Table 4.

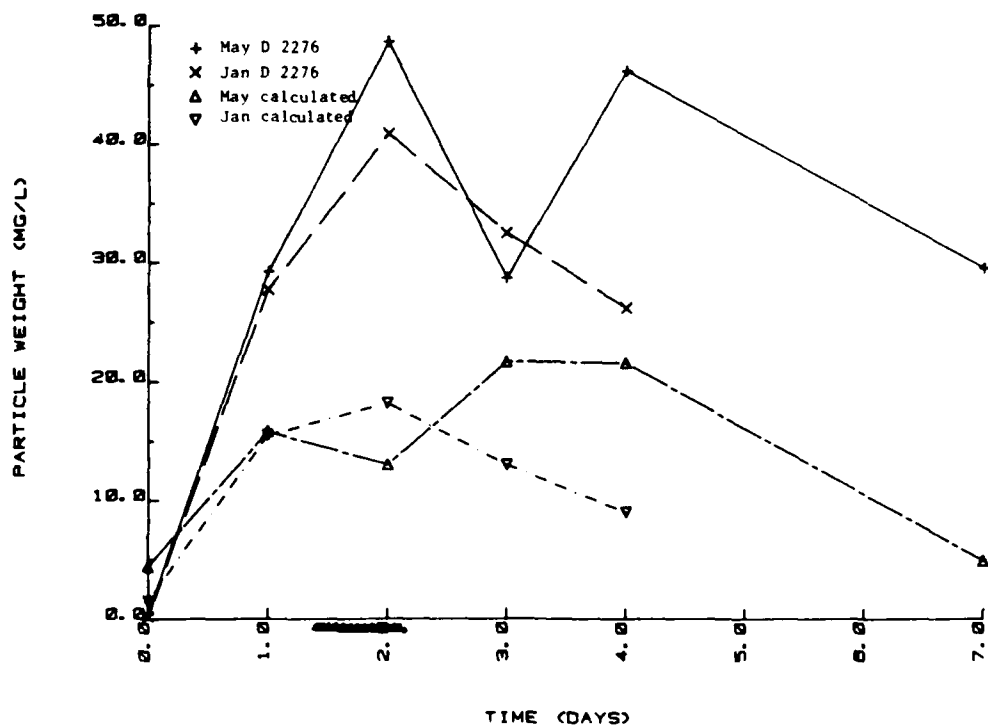


FIGURE 13. COMPARISON OF CALCULATED AND MEASURED PARTICULATE WEIGHT FOR ADDITIVE-FREE CAT 1-H AGED AT 95°C

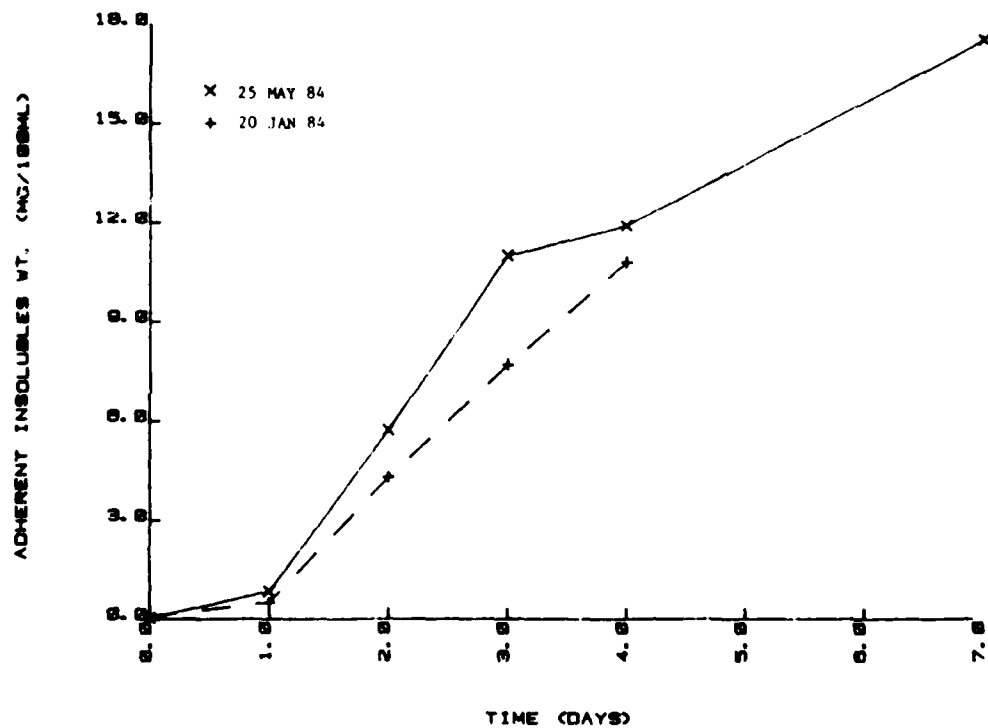


FIGURE 14. ADHERENT INSOLUBLES FORMATION VERSUS TIME FOR ADDITIVE-FREE CAT 1-H AGED AT 95°C

3. Neat Cat 1-H Fuel at 80°C

This category contains the most extensive variety of experimental containers; 1-liter Pyrex® bottles, 12-gallon Pyrex® carboy, and 30-gallon metal drum. Duplicate experiments using the 12-gallon (45-liter) container yielded calculated and gravimetric values that corresponded reasonably well. A comparison of data from simultaneous testing of the 12-gallon (45-liter) Pyrex® container and the 30-gallon (112.5-liter) metal drum also indicated minimal variation in both D 2276 and calculated data. These data are represented in Figures 15 through 17. Figure 18 compares the rate of adherent gum formation for a neat fuel with fuels to which Additive-15, Additive-11, and the SPA have been added. The reduction of adherent insolubles formation in the additive-treated fuels is quite significant. Elemental data may be found in Table 4.

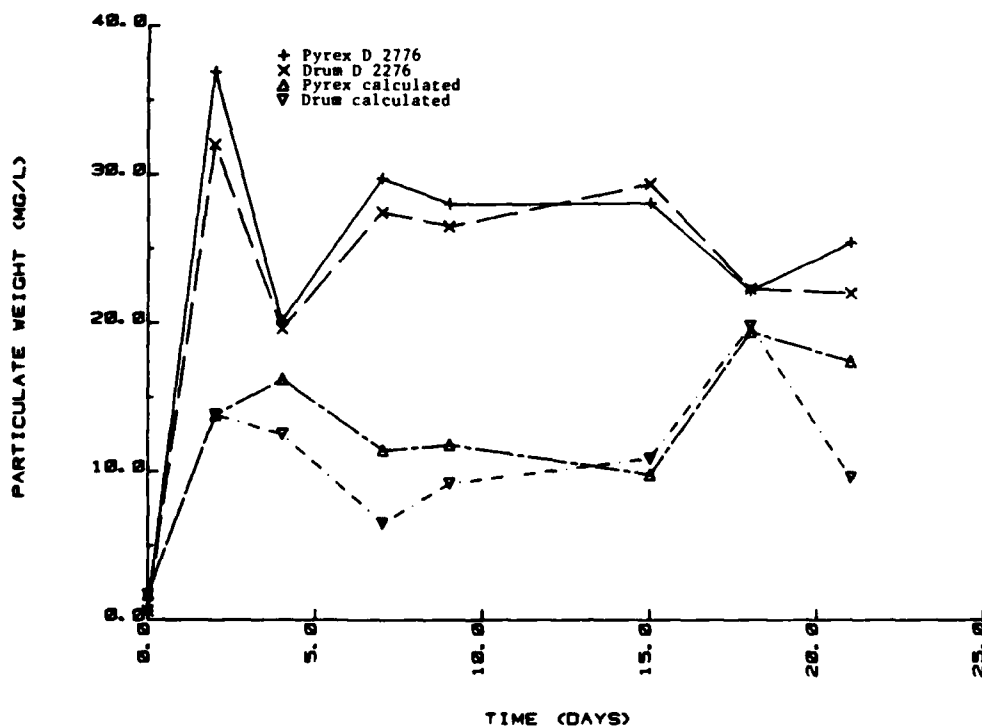


FIGURE 15. COMPARISON OF CALCULATED AND MEASURED PARTICULATE WEIGHT FOR ADDITIVE-FREE CAT 1-H AGED AT 80°C

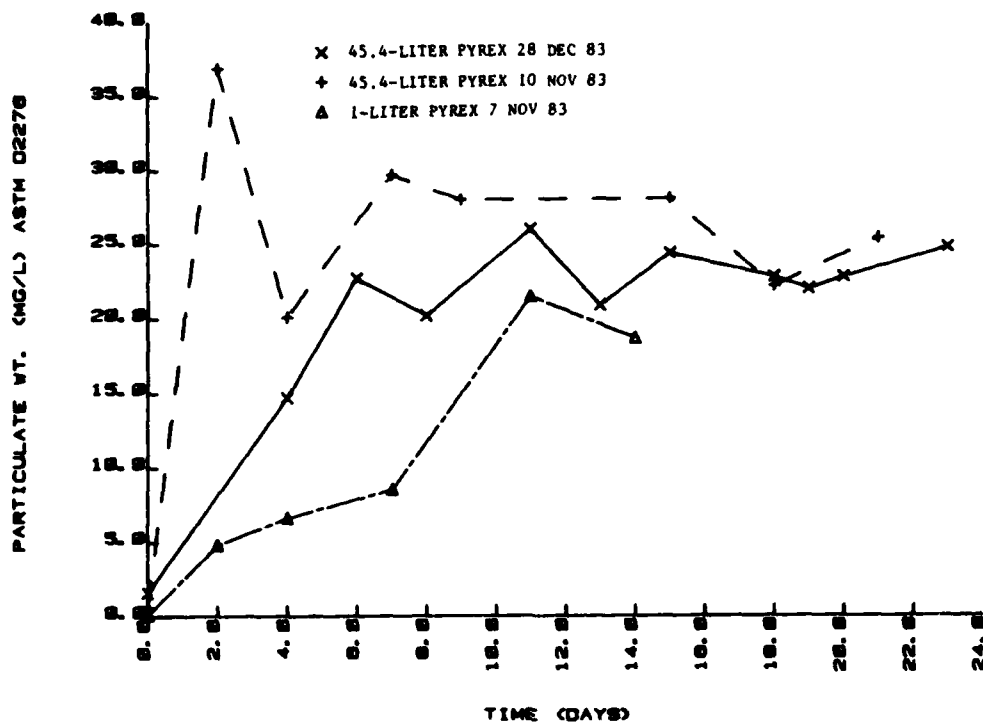


FIGURE 16. MEASURED PARTICULATE WEIGHTS VERSUS TIME FOR ADDITIVE-FREE CAT 1-H AGED AT 80°C IN PYREX® CONTAINERS

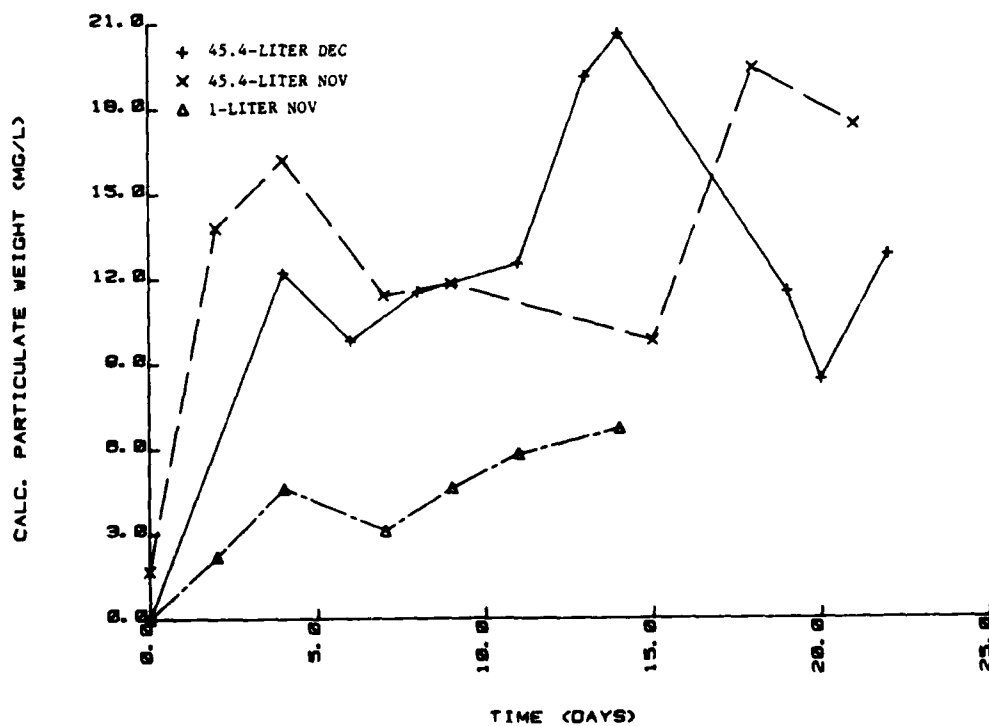


FIGURE 17. CALCULATED PARTICULATE WEIGHT AS A FUNCTION OF TIME FOR ADDITIVE-FREE CAT 1-H AGED AT 80°C IN PYREX® CONTAINERS

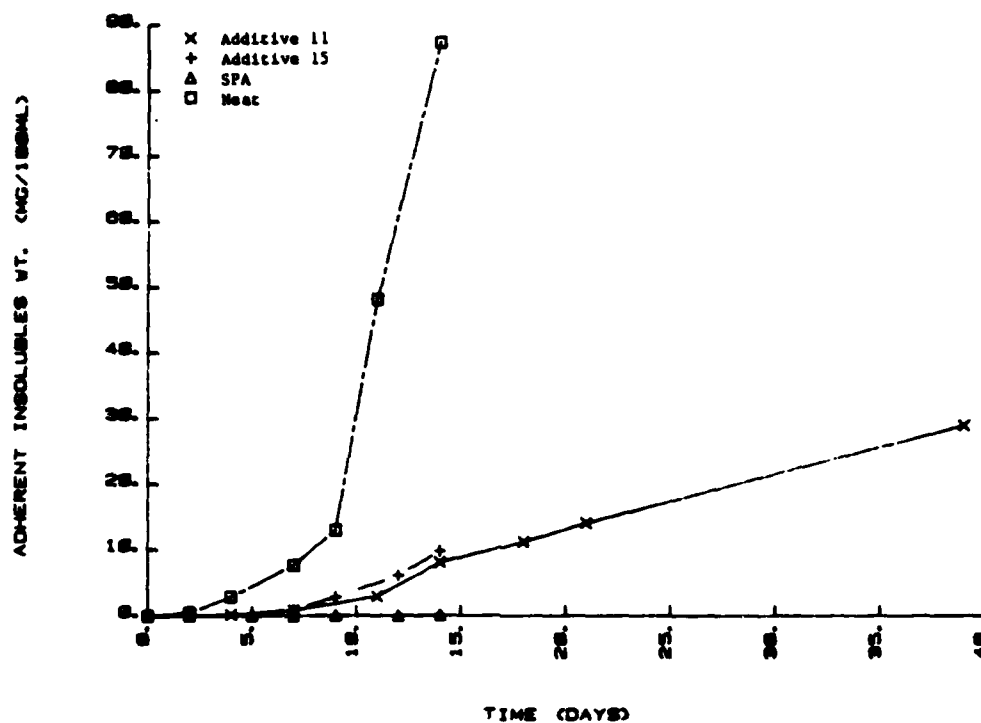


FIGURE 18. COMPARISON OF ADHERENT INSOLUBLES FORMATION FOR CAT 1-H AGED AT 80°C WITH VARIOUS ADDITIVES PRESENT

4. Neat Cat 1-H Fuel at 65°C

The 5-month period (Dec. 1983-May 1984) between tests again allowed a time-dependent measure of repeatability. Except for one point at 7 days in the D 2276 data, all values repeated well. Adherent insolubles weight appeared to be higher by an average factor of about 3 for the older sample. The induction period of 4 days is the same for both experiments. These data are given in Figures 19 and 20. Elemental data are given in Table 4.

5. Cat 1-H With Additive-15 + Additive-B and Single-Package Additive

In each of the tests performed, values obtained for the single-package additive (SPA) were essentially the same as or less than the values obtained for the Additive-15 treated fuel. The tests included filterable particulate weight by ASTM D 2276, calculated particulate weight, adherent insolubles weight, and peroxide number (see also Cat 1-H + Additive-11). The data

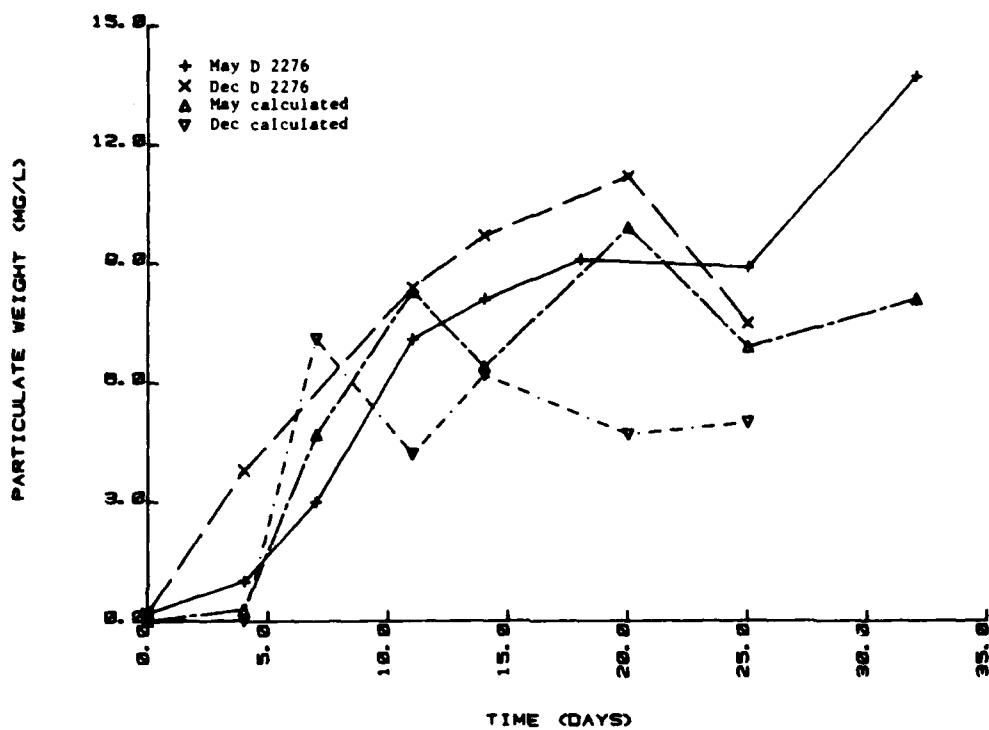


FIGURE 19. COMPARISON OF CALCULATED AND MEASURED PARTICULATE WEIGHT FOR ADDITIVE-FREE CAT 1-H AGED AT 65°C

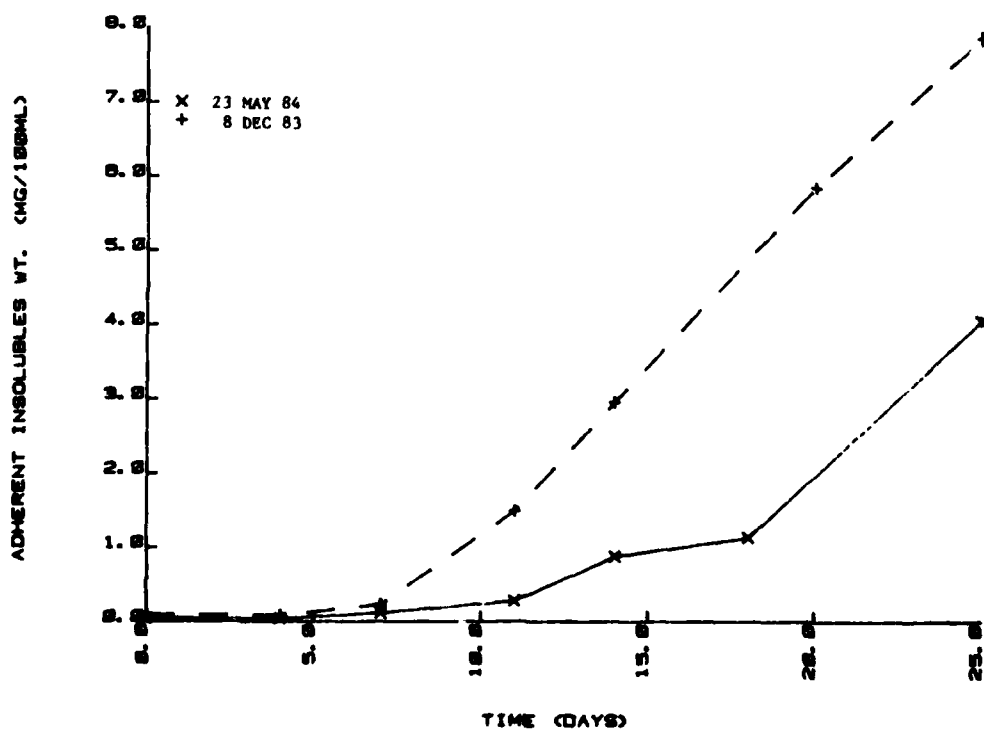


FIGURE 20. ADHERENT INSOLUBLE FORMATION VERSUS TIME FOR ADDITIVE-FREE CAT 1-H AGED AT 65°C

generated are shown in Figures 21 and 22. Insufficient adherent insolubles were formed to allow determination of N or S composition in the SPA-treated fuel. A value for %N in adherent insolubles from the Additive-15/Additive-B test is given in Table 4. A comparison of adherent insolubles formed from Additive-11, Additive-15, SPA-treated, and neat Cat 1-H is shown in Figure 18.

IV. CONCLUSIONS

1. It is possible to model mathematically the ASTM D 2276 gravimetric filterable particulate analysis using particle size and number distribution data.
2. The assumption of a spherical geometry for the particulates may be incorrect because of the consistently greater results for gravimetric data. The difference factor is greater than could be reasonably expected from a density variation (an increase from 2 to 10 times the assumed density would be required for data to be equivalent within a factor of 2).
3. Measurable quantities of fuel-insoluble particulate matter generally form before measurable quantities of adherent fuel insoluble materials, indicating that particulate matter is present as a possible precursor to adherent insolubles.
4. The weight of particulate matter formed in smaller containers appears less than that formed in larger containers, which could indicate an inverse relationship to the contact surface area/volume ratio. Adherent insolubles formation was not monitored in the larger containers as part of this program. A relative increase in adherent insolubles weight in the smaller containers would verify Item 3 above.
5. Use of fuel additives appeared to reduce the quantity of filterable particulate matter. The dispersants employed in the additives may have caused particle size to be maintained below detection limits of the ex-

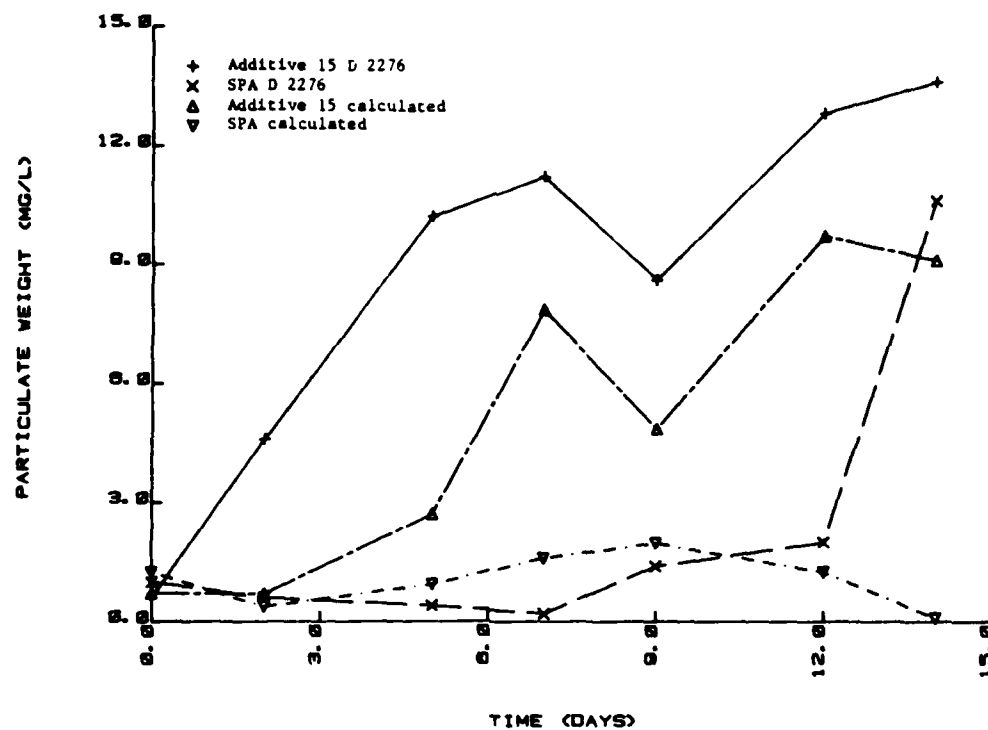


FIGURE 21. COMPARISON OF CALCULATED AND MEASURED PARTICULATE WEIGHT FOR ADDITIVE-TREATED CAT 1-H

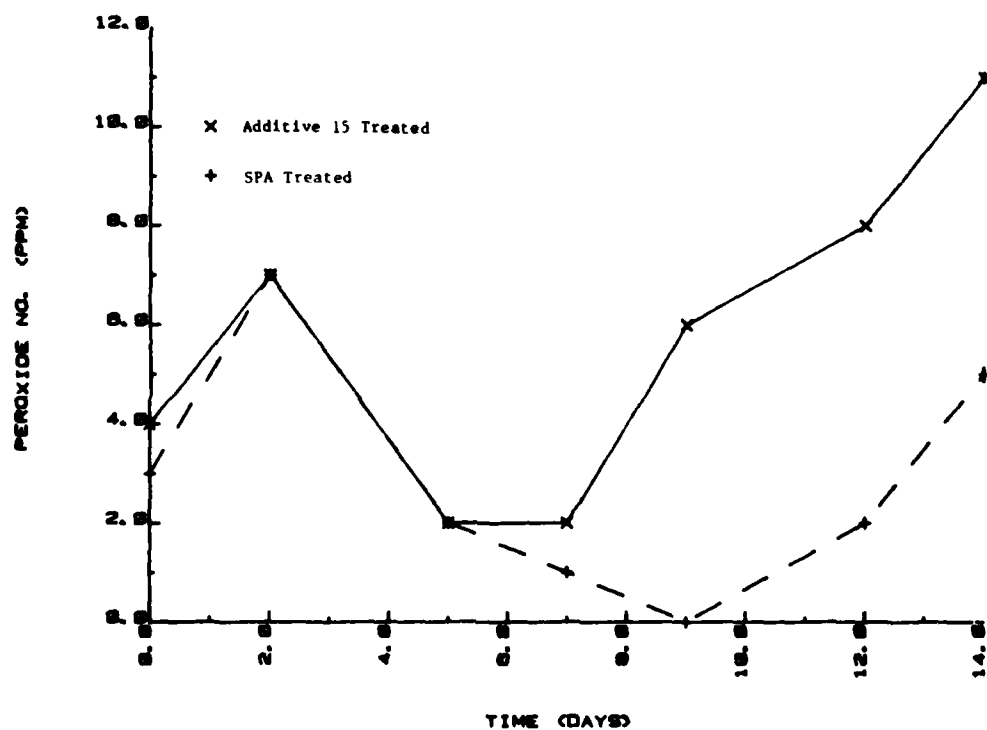


FIGURE 22. PEROXIDE NUMBER VERSUS TIME FOR ADDITIVE-TREATED CAT 1-H AT 80°C

perimental procedure. Conversely, the quantity of adherent insolubles formed appears greater in the additive-treated fuel tests. This relationship may further verify item 3 above.

6. Repeat experiments up to 5 months apart provide similar data from the same fuel. This indicates that ambient drum storage does not materially affect fuel stability even through the winter/spring period. One exception was adherent insoluble formation being reduced by a factor of 3 in the second test.
7. An additive-treated fuel aged at three separate temperatures (15°C intervals) produced adherent insolubles in proportionately higher quantities. The particulate matter formation at the intermediate temperature did not increase in the anticipated amount, possibly due to slow formation rates or detection limits being too high to properly measure the actual quantity of insoluble particulate matter formed.
8. Nitrogen and sulfur-containing chemical species are major factors in fuel-insoluble product formation. The concentration of these species in the adherent insolubles can be many orders of magnitude higher than in the fuel. Nitrogen is more pronounced in this respect than sulfur.
9. Although values for peroxide number reflect changes as the fuel is aged, interpretation of these results is difficult because of their erratic nature.
10. Bromine number, reflecting olefinic bonding, remains constant, indicating a lesser role for double bonds in the overall reaction sequence than expected.
11. Natural sunlight, even through reflection, has a major effect on fuel deterioration studies.

V. RECOMMENDATIONS

Recommendations for continuing investigation into the mechanism of middle distillate fuel determinations and additive inhibitors are:

1. Investigate the role of dissolved oxygen on particulate and adherent gum growth through isotopic labeling ($^{18}\text{O}_2$).
2. Chemically cleave the highly polar/nonvolatile portions of the deterioration products to allow passage through GC columns for identification purposes.
3. Investigate/employ pyrolysis techniques for volatilization/identification of the deterioration products.
4. Place a greater emphasis on the isolation/identification of existent gums and their role in fuel insoluble gum formation. Relate these findings to studies in other laboratories using single-component systems.
5. Continue energy of activation studies and relate findings to fuel and gum compositions.
6. Investigate adherent insoluble formation as a function of surface area/fuel volume ratio to determine the degree to which wall effects influence insoluble product formation.

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